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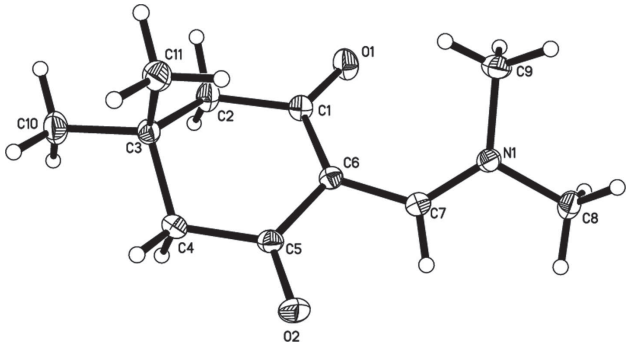
Crystal structure of 2-((dimethylamino)methylene)-5,5-Dimethylcyclohexane-1, 3-dione, $C_{11}H_{17}NO_2$



DOI 10.1515/ncrs-2016-0002
Received February 15, 2016; accepted March 4, 2016; available online April 2, 2016

Abstract

$C_{11}H_{17}NO_2$, monoclinic, $P2_1/c$ (no. 14), $a = 5.9245(2)$ Å, $b = 17.7243(6)$ Å, $c = 10.2915(4)$ Å, $\beta = 95.352(1)°$, $V = 1075.97(7)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0419$, $wR_{ref}(F^2) = 0.116$, $T = 100(2)$ K.

CCDC no.: 1444949

In the figure the title molecule is shown.

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Table 1: Data collection and handling.

Crystal:	Yellow blocks, size $0.341 \times 0.287 \times 0.254$ mm
Wavelength μ	Mo K_α radiation (0.71073 Å) 0.82
Diffractometer, scan mode:	Bruker Apex-II, φ and ω
$2\theta_{max}$, completeness,	67,546°, >99 %,
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	29627, 4312, 0.0386
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3426
$N(param)_{refined}$:	131
Programs:	SHELX [14], Bruker programs [15]

Source of material

A mixture of 5,5-dimethylcyclohexane-1,3-dione (1.4 g, 0.01 mol) and dimethylformamide-dimethylacetal (DMF-DMA) (1.19 g, 0.01 mol) in dry xylene (20 mL) was refluxed for 2 h, the reaction mixture was cooled, and the obtained solid was recrystallized from ethanol to give the corresponding 2-(dimethylamino)methylene)-5,5-dimethylcyclohexane-1,3-dione [13]. Yield%: 71, m.p. = 94°C, IR, cm^{-1}: 2934, 2855 cm^{-1} (CH aliph.), 1663 cm^{-1} (C = O). ^{1}H-NMR (DMSO-*d*6, ppm): 0.96 [s, 6H, $2CH_3$], 2.21 [s, 4H, $2CH_2$], 3.04 [s, 3H, N—CH_3], 3.38 [s, 3H, N—CH_3], 7.95 [s, 1H, CH]. MS (EI, 70 EV): m/z = 195 [M$^+$]. Anal. Calcd for $C_{11}H_{17}NO_2$(195.26): C, 67.66; H, 8.78; N, 7.17. Found: C, 67.32; H, 9.05; N, 7.46.

Experimental details

Cell refinement and data reduction were carried out by Bruker SAINT [15].

Discussion

The miscellaneous biological activities of 2-((dimethylamino) methylene)-5,5-dimethyl-cyclo-hexane-1,3-dione offshoots as well as their vital solicitations in pharmaceutical productions have enthused research in this expanse [1–4]. Cyclohexane- 1,3-diones are significant, distinguishing molecular parts mutual for herbicides [5, 6]. The inhibitory effect of these products are a consequence of their aptitude to make complexes with Fe++ in the vigorous location of the enzyme. Cyclohexane-1,3-dione well known as ancestors of

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.53247(11)	0.27033(4)	0.65318(6)	0.02193(14)
O2	0.99206(11)	0.48828(4)	0.67490(6)	0.02170(14)
N1	0.50728(11)	0.39307(4)	0.44148(7)	0.01520(13)
C1	0.66141(14)	0.31608(4)	0.71258(8)	0.01516(14)
C2	0.76024(15)	0.29583(5)	0.85019(8)	0.01908(16)
H2A	0.8859	0.2596	0.8437	0.023*
H2B	0.6417	0.2698	0.8952	0.023*
C3	0.84918(13)	0.36225(5)	0.93435(8)	0.01558(14)
C4	1.01353(14)	0.40577(5)	0.85681(8)	0.01921(16)
H4A	1.0655	0.4511	0.9072	0.023*
H4B	1.1480	0.3737	0.8477	0.023*
C5	0.91678(13)	0.43029(5)	0.72275(8)	0.01526(14)
C6	0.73853(13)	0.38513(4)	0.65467(7)	0.01379(14)
C7	0.68621(13)	0.40646(4)	0.52283(8)	0.01431(14)
H7A	0.8004	0.4355	0.4872	0.017*
C8	0.51306(15)	0.40943(5)	0.30210(8)	0.01861(16)
H8A	0.6566	0.4342	0.2880	0.028*
H8B	0.3866	0.4428	0.2728	0.028*
H8C	0.4998	0.3622	0.2524	0.028*
C9	0.28943(14)	0.36555(6)	0.47784(10)	0.02474(19)
H9A	0.2837	0.3711	0.5722	0.037*
H9B	0.2719	0.3122	0.4540	0.037*
H9C	0.1665	0.3948	0.4317	0.037*
C10	0.97460(17)	0.33525(6)	1.06268(9)	0.02401(18)
H10A	1.1038	0.3038	1.0440	0.036*
H10B	0.8712	0.3056	1.1115	0.036*
H10C	1.0292	0.3790	1.1147	0.036*
C11	0.64950(15)	0.41210(5)	0.96509(9)	0.02109(17)
H11A	0.5704	0.4307	0.8835	0.032*
H11B	0.7061	0.4550	1.0188	0.032*
H11C	0.5444	0.3825	1.0127	0.032*

impending anti-diabetic drugs [7]. In continuation of our synthesis of biologically active heterocyclic compounds [8–12], we report herein the results of the synthesis and reactions of 5,5-dimethylcyclohexane-1,3-dione with dimethylformamide-dimethylacetal (DMF-DMA).

Thus, interaction of 5,5-dimethylcyclohexane-1, 3-dione with dimethylformamide-dimethylacetal (DMF-DMA) in dry xylene afforded the corresponding 2-(dimethylamino) methylene)-5,5-dimethylcyclohexane-1,3-dione. The structure of this compound was established on the basis of elemental analysis, IR, ^{1}H-NMR, mass spectral data and X-ray analysis (figure, table 1 and 2).

Acknowledgements: The authors would like to extend their sincere appreciation to the Deanship of Scientific Research at King Saud University for funding of this research through the Research Group Project no. **RGP-VPP- 302**.

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